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EVALUATION OF LOW-PRESSURE DROP ANTIMICROBIAL AND HYBRID AIR FILTERS

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EXECUTIVE SUMMARY

A. Objective

The purpose of this research was to perform a competitive evaluation of low-pressure-drop antimicrobial particle filter media.

B. Background

Traditional purification of aerosol-contaminated air streams has been performed by mechanical filtration. Existing particle filters will stop bacterial and viral penetration to an extent determined by filtration efficacy, and that efficacy is related to pressure drop. There is a need to develop filters that provide protection from bio-aerosols at a reduced pressure drop. One example of this need is that many ships do not have protection from Biological Warfare (BW) agents, and are not easily retrofitted due to existing HVAC limitations. One possible interim solution is the deployment of a temporary ship intake filter of low pressure drop, while still maintaining high filtration efficiency for bioaerosols.

A promising approach to increasing the efficacy of low-pressure-drop filters against bio-agents is the use of Self-Decontaminating Materials (SDMs). Available air filters based on SDMs were evaluated to determine the current status of these technologies.

Test methodologies for determining the performance of SDM filters against aerosol threats were sought through literature search and collaboration. Physical and chemical properties, and bio-filtration efficiency of SDMs under current Tech Base development (I₃ resin, *N*-halamines, and quaternary amines) and COTS filters (*e.g.*, Ag/Ag⁺) were evaluated.

C. Scope

This project was funded for the initial year of a planned three-year effort. A literature review was conducted to identify available commercial-off-the-shelf (COTS) SDM filter media. Samples of these COTS materials were obtained from the manufacturers and in-house SDMs were prepared. Screening methods were evaluated to determine antimicrobial activity of the SDM filter media. Limited testing of SDMs against bio-aerosols was conducted.

D. Methods and Procedures

Methods developed by the American Association of Textile Chemists and Colorists (AATCC) for testing antimicrobial fabrics (AATCC methods 100 & 147) were evaluated for use on the SDM filter media. The AFRL bioaerosol test chamber located at Tyndall AFB, building 9768 was used to challenge the media in a filtration test with a bio-aerosol.

E. Results

Media based on an iodinated polymeric resin, an *N*-halamine, and a quaternary ammonium salt all showed significant biocidal activity. A silver-based medium was ineffectual. The same media that gave positive results in the screening tests also showed enhanced protection in preliminary bioaerosol testing.

F. Conclusions

AATCC methods 100 and 147 were found to be suitable for screening SDM filter media for antimicrobial activity. The screening method performance was predictive of the performance in bioaerosol tests. The media based on the iodinated polymeric resin, the quaternary ammonium salt, and the *N*-halamine merit further bioaerosol testing.

G. Recommendations

New SDM air filtration media can be screened with AATCC method 147 as an indication of possible efficacy against bioaerosols. Recommend an in-house program be established to further advance SDM air filtration media, as multiple candidates for the deployment of this technology exist, and government expertise is needed to guide future acquisition programs.

PREFACE

This technical report describes work performed by Applied Research Associates personnel from March 2003 to January 2004 under Scientific Engineering and Manpower Assistance Support (SEAMAS) Contract No. F08637-03-C-6006 to AFRL/MLQ, Tyndall Air Force Base, Florida. Support for this project was funded by Defense Threat Reduction Agency (DTRA), project number CP20002. The AFRL work unit manager was Patrick Sullivan.

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1.0 Introduction

1.1 Objective

The purpose of this research was to evaluate low-pressure-drop (low- ΔP) antimicrobial particle filter materials currently available, and to develop new and/or hybrid low- ΔP antimicrobial particulate filter materials.

1.2 Background

Traditional purification of aerosol-contaminated air streams has been performed by mechanical filtration. Existing particulate filters will stop bacterial and viral penetration to an extent determined by filtration efficacy, and that efficacy is related to pressure drop (ΔP). There is a need to develop filters that provide protection from bioaerosols at lower ΔP .

A promising approach to increasing the efficacy of low- ΔP filters against bioagents is the use of self-decontaminating materials (SDMs). Available air filters based on SDMs were evaluated to determine the current status of these technologies.

AFRL/MLQL has been developing SDMs for such non-filtration applications as Battle Dress Uniforms, CBD barrier materials and shelter fabrics for about five years. One focus of activity at AFRL is a class of compounds called *N*-halamines (Worley 2005). Other SDMs under development elsewhere supported by Defense Threat Reduction Agency (DTRA) funding include an I_3 resin and quaternary amines.

Test methodologies for determining the performance of SDM filters against aerosol threats were sought through literature search and collaboration. The results of this search are listed in Appendix A.

1.3 Scope

The tasks of the project included performing a survey of currently available COTS filter materials, obtaining COTS filter samples, preparing or obtaining samples of filter materials under development within government labs (and private labs where available), evaluating methods to screen the filter materials, and performing bioaerosol tests on the filter materials. However, this project was funded for the initial year of a planned three-year effort, which limited the effort to the initial set of SDM filter media and eliminated second-generation filter materials and the production of additional hybrid filter materials.

2.0 Methods and Procedures

2.1 Equipment

For the screening tests, standard microbiological testing supplies such as Mueller–Hinton agar plates, cotton swabs, etc. were used.

For the bioaerosol tests conducted at AFRL/MLQL, an aerosol chamber designed specifically for applying bioaerosol challenges to candidate reactive materials was used. The BioAerosol Test System (BATS, Figures 1 and 2) is a port-accessible aerosolization chamber communicating with a temperature-controlled mixing plenum and thence to a sampling plenum supplying a homogeneous aerosol to six sampling ports. Three six-jet Collison nebulizers (BGI Inc, Waltham, Mass.) deliver a mist of mean diameter $\sim 2\ \mu\text{m}$ into the mixing plenum to create the bioaerosols. Air is drawn into a central vacuum line along a path from the sampling plenum through parallel lines of PVC tubing (Excelon® RNT, US Plastics, Lima, Ohio). Each path runs through a test article and thence through one AGI-30 all-glass impinger (Chemglass, Vineland, N.J.) partially filled with a liquid collection medium. The volume of air passing in each path is controlled by a mechanical flow meter (Blue–White 400, Huntington Beach, California, or PMR1-101346, Cole–Parmer, Vernon Hills, Illinois). At the end of the sampling path, the air exhausts through a conventional high-efficiency particle-arresting (HEPA) filter and the vacuum pump that drives the air movement.

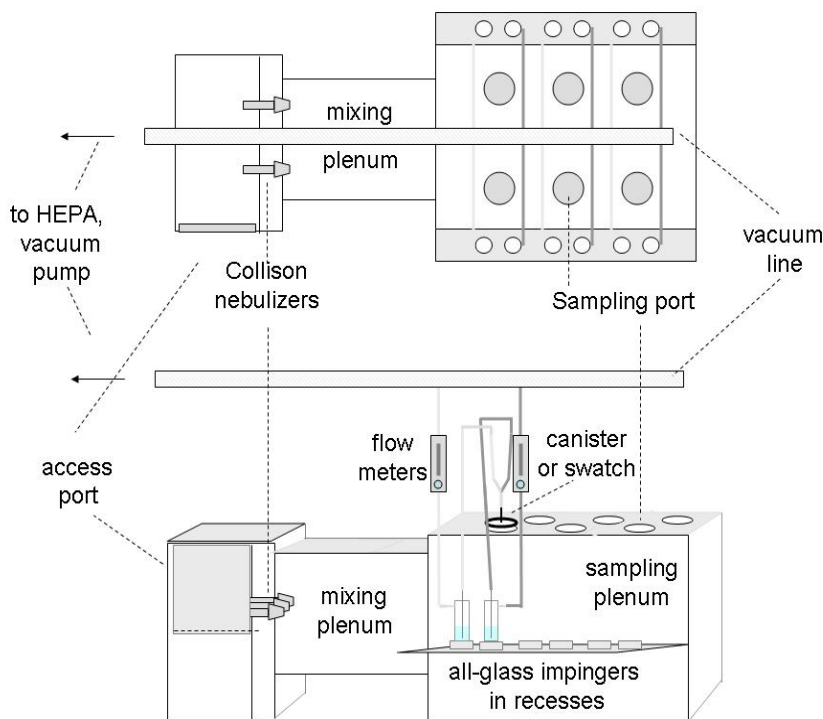


Figure 1. Top and Side View of BioAerosol Test System (BATS) Components.



Figure 2. AFRL Bio-Aerosol Test System (BATS).

2.2 Procedure

2.2.1 Material Selection and Preparation

SDM filter media based on four different chemistries were selected for testing: silver, *N*-halamines, I₃ resin, and quaternary amines. The silver and I₃ resin media were obtained from the manufacturers.

For the other two chemistries, a substrate was needed to create an air filter medium. Lydall, Inc., a manufacturer of non-woven filter media used in HEPA filters, provided samples of HEPA-grade media and near-HEPA-grade media onto which the *N*-halamine and quaternary amine treatments were applied. Lydall numbers 2991 and 3242 were used.

2.2.2 Screening Tests

AATCC Method 147 (AATCC 2004) and Method 100 (AATCC 2004) were evaluated as well as using the Kirby–Bauer Method (Amin 2006).

2.2.3 Bio-aerosol Tests

Flat sheet material supplied by AFRL/MLQL laboratories were cut into 4.7-cm diameter discs and sealed by compression into an *O*-ring on the swatch holder (figure 3); the integrity of this seal was not tested. The swatch holder was then plumbed to a sampling port. All sets of measurements included a positive control, a port which was sampled with no obstruction in the airway. The number of plaque-forming units (PFUs) measured

through this port was reported as the challenge level. MS2 coli phage (ATCC 15597-B1) stock was diluted to $\sim 2 \times 10^8$ PFU/mL in sterile water and delivered to three six-jet Collison nebulizers. Compressed air (20 psi) was fed into the nebulizers to deliver a uniform microorganism–air aerosol that was drawn into the plenum box of the BATS and thence through the test articles and positive control. The air flow through each swatch holder was 5.4 LPM and the total test time was 1 hour.

A standard plaque assay was used to determine phage concentrations of the samples and the positive control. Each sample was serially diluted 1:10 out to 10^{-4} and 1-mL aliquots of each dilution were used in the plaque assay as follows: One mL of the test solution was mixed with 1 mL of *Escherichia coli* (ATCC 15597) culture (grown to mid log-phase) and 9 mL of MS2 medium (1% tryptone, 0.1% yeast extract, 0.8% sodium chloride, and 0.1% dextrose, 1% agar), held at 55 °C. The solution was mixed three times then poured into sterile Petri dishes. The Petri dishes were incubated overnight at 37 °C and plaques were counted the following day. Total PFU counts were determined by averaging the PFUs determined on the triplicate sample plates, and then multiplying by the dilution factor and by the impinger volume.



Figure 3: Swatch Holders

3.0 Results and Discussion

3.1 Screening Test Results

AATCC Methods 100 and 147 gave mutually consistent indications of antimicrobial activity in these tests. After a few initial trials, Method 100 was not pursued further, due to its being more onerous to perform than Method 147. Although Method 100 is more quantitative than the agar-plate methods, aerosol testing is required to determine the effectiveness of air filtration media.

The Kirby–Bauer method worked equally as well as Method 147 in identifying antimicrobial activity, and is preferred because the sample of filter medium is smaller, so multiple tests can be conducted in one agar plate.

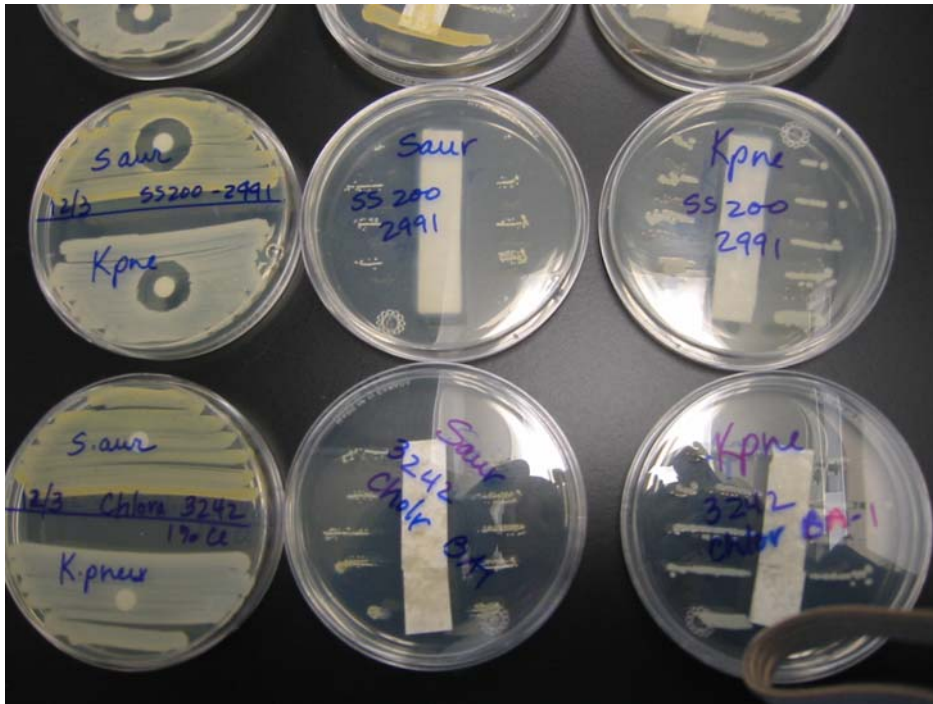


Figure 4. AATCC Method 147 and Kirby–Bauer Method for N-halamine and Quaternary Amine Media



Figure 5. Kirby–Bauer Method for Silver and I₃ Resin Media

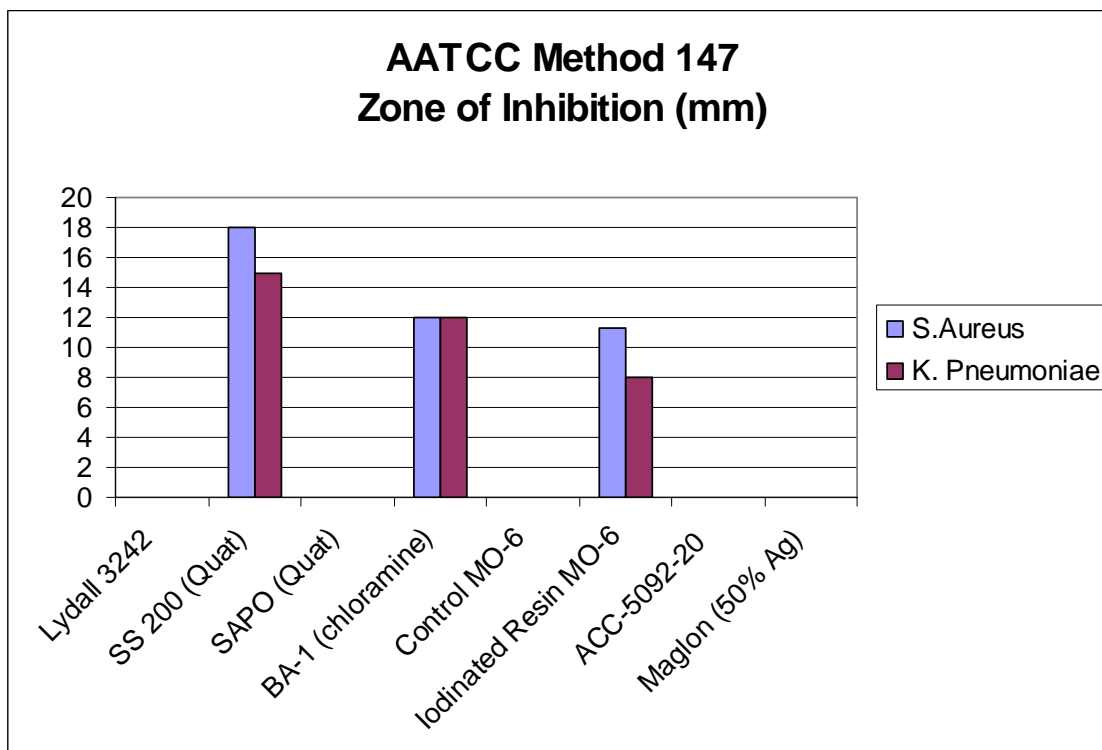


Figure 6. AATCC Method 147 Summary of Results for SDM Filter Media. Lydall 3242 is the untreated substrate (control).

In general, the I₃ resin, quaternary amines, and *N*-halamines displayed comparable levels of antimicrobial activity in the screening tests, all showing significant zones of inhibition around the samples. The silver media at 10%, 25%, and 50% loading of Ag by weight all showed no antimicrobial activity. Some inconsistency was evident in the AFRL-prepared samples—a fraction of the replicates had no activity for both the *N*-halamine and quaternary amine treatments. This was interpreted to be a result of problems in sample preparation, and not representative of the effectiveness of the treatment itself. All of the samples were thoroughly rinsed after treatment to avoid leaching during the tests.

3.1 Bioaerosol Test Results

As shown in Table 1, both the *N*-halamine and quaternary amine treatments showed a dramatic improvement in biological filtration efficiency of MS2 coli phage compared to

Table 1. Bioaerosol Test Results

Challenge of HEPA material with MS2 coli phage (1 hour @ 5.4 L/min)	
Samples	Viable penetration of MS2 coli phage
Challenge	3.00 E+06 PFU
Untreated HEPA	1.40 E+04
SM 051305A Quat & HEPA	0*
SM 051305G TTDD sol. & HEPA	0*
* detection limit = 3.3 PFU	

control, essentially eliminating all viable penetration of the challenge. The I₃ resin was not tested as 47-mm disks due to sample availability, but showed similar results when tested as an assembled canister.

4.0 Conclusions

AATCC methods 100 and 147 were found to be suitable for screening SDM filter media for antimicrobial activity. Method 147 is preferred since it is adequate for screening and much easier to perform than Method 100. Small sample disks (5 mm), as in the Kirby–Bauer method, can be substituted for the normal swatch used in Method 147. The screening method performance was predictive of the performance in bioaerosol tests. Media based on the iodinated polymer resin, the quaternary ammonium salt and the *N*-halamine all proved effective in enhancing protection during bioaerosol testing and merit further investigation. The failure of the silver-treated materials is not explained.

5.0 Recommendations

Recommend an in-house program be established to further advance SDM air filtration media, as multiple candidates for the deployment of this technology exist, and government expertise is needed to guide future acquisition programs.

6.0 References

AATCC, (2004). AATCC Test Method 100-2004. Antibacterial Finishes on Textile Materials: Assessment of. Research Triangle Park, N.C.

AATCC, (2004). AATCC Test Method 147-2004. Antibacterial Activity Assessment of Textile Materials: Parallel Streak Method. Research Triangle Park, N.C.

Amin, J. L., Sarah; Owen, Angela. (2006). "Kirby–Bauer Method." from <http://aminj.myweb.uga.edu/KIRBY-BAUER.html>.

Worley, S. D. C., Yongjun; Wang, Jia-Wang; Wu, Rong; Li, Yanzun (2005). *N*-halamine siloxanes for use in biocidal coatings and materials. USA, Vanson Halosource, Inc. (Redmond, Washington). US Patent# **6,969,769**.

Appendix A: Literature Search

“Testing of Laminar Air Flow Units by Particle Counting and by Microbial Methods,” Bernt Aslund, Olof T. Olson *Acta Pharm. Suec.* **13**: 469–474 (1976).

Method for Measuring the Spatial Variability of Aerosol Penetration through Respirator Filters, Cuiling Huang, Klaus Willeke, Ying Qian, Sergey Grinshpun, Vidmantas Ulevicus *American Industrial Hygiene Journal* **59**:461–465 (1998).

“Aerosol Penetration through Surgical Masks,” Chih–Chieh Chen, Klaus Willeke *AJIC* August 1992 pp177–184

“Collection of Three Bacterial Aerosols by Respirator and Surgical Mask Filters under Varying Conditions of Flow and Relative Humidity,” N.V. McCullough, L.M. Brosseau and D. Vesley *Ann Occup. Hyg.* **41** [6]: 677–690 (1997).

“Mycobacterial Aerosol Collection Efficiency of Respirator and Surgical Mask Filters under Varying Conditions of Flow and Humidity,” Lisa M. Brosseau, Nicole Vars McCullough, David Vesley *Appl. Occup. Environ. Hyg.* **12** [6]: 435–445 (1977).

“Assessment of Air Filters Treated with a Broad Spectrum Biostatic Reagent,” D.L. Price, R.B. Simmons, D.L. Ramey, S.A. Crow, D.G. Ahearn *Proceedings of Indoor Air* **6**: 527–532 (1993).

“Efficacy of Antimicrobial Filter Treatments,” Karin K. Foarde, James T. Hanley, Alan C. Veeck *ASHRAE Journal* 52-58 (2000)

“Determine the Efficacy of Antimicrobial Treatments of Fibrous Air Filters,” Karin K. Foarde, James T. Hanley *ASHRAE Transactions: Research* **4429** (RP-909): 156–170.

“Development of a Method for Measuring Single-Pass Bioaerosol Removal Efficiencies of a Room Air Cleaner,” Karin K. Foarde, James T. Hanley, David S. Ensor, Peter Roessler *Aerosol Science & Technology* **30**:223–234 (1999).

METHODS:

AATCC Test Method 100-1999 *Antibacterial Finishes on Textile Materials: Assessment of*

ANSI/ASHRAE Standard 52.1-1992 *Gravimetric and Dust-Spot Procedures for Testing Air-Cleaning Devices Used in General Ventilation for Removing Particulate Matter*

ANSI/ASHRAE Standard 52.2-1999 *Method of Testing General Ventilation Air Cleaning Devices for Removal Efficiency by Particle Size*

ASTM D 1505-85 (1990) *Standard Test Method for Density of Plastics by the Density-Gradient Technique*

ASTM D 6329-98 *Standard Guide for Developing Methodology for Evaluating the Ability of Indoor Materials to Support Microbial Growth Using Static Environmental Chambers*

ASTM F 778-88 (1993) *Standard Method for Gas Flow Resistance Testing of Filtration Media*

ASTM F 872-84 (1990) *Standard Specification for Filter Units, Air Conditioning: Viscous Impingement Type, Cleanable*

ASTM F902-84 (1990) *Standard Practice for Calculating the Average Circular-Capillary –Equivalent Pore Diameter in Filter Media from Measurements of Porosity and Permeability*

ASTM F1040-87 (1993) *Standard Specification for Filter Units, Air Conditioning: Viscous Impingement and Dry Types, Replaceable*

ASTM F1215-89 *Standard Test Method for Determining the Initial Efficiency of a Flatsheet Filter Medium in an Airflow Using Latex Spheres*

ASTM F1471-93 *Standard Test Method for Air Cleaning performance of a High-Efficiency Particulate Air-Filter System*

ASTM F2101-01 *Standard Test Method for Evaluating the Bacterial Filtration Efficiency of Medical face Mask Materials, Using a Biological Aerosol of Staphylococcus aureus*

ASTM G21-90 *Standard Practice for Determining Resistance of Synthetic Polymeric Materials to Fungi*

ASTM G22-76 (1990) *Standard Practice for Determining Resistance of Plastics to Bacteria*

DOE-STD3025-99 *Quality Assurance Inspection and Testing of HEPA Filters*

IES-RP-CC-007-1.0 *Testing of ULPA Filters*

IES-RP-CC-021 *Testing of HEPA and ULPA Filter Media*

IES-RP-CC-001-86 *HEPA Filters*

MIL SPEC MIL-F-51079D March 1985 as amended February 1988 *Military Specification for Filter Medium, Fire Resistant, High Efficiency*

NIOSH Manual of Analytical Methods Method 0800, Issue 1 15 January 1998
Bioaerosol Sampling (Indoor Air)

TAPPI T 487 cm-1993 *Fungus Resistance of Paper and Paperboard*

UL-586 *Standard for High Efficiency, Particulate, Air Filter Units*

UL-900 *Standard for Test Performance of Air Filter Units*